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Water transport in freshly-mixed mortars containing cellulose ethers

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1. Introduction

Polysaccharides are polymers frequently introduced into mortar formulations in order to improve the workability and water retention capacity of the fresh materials [1]. Among all the polysaccharides, cellulose ethers seem to be the most suitable molecules to obtain a mortar with pretty good water retention ability (*i.e.* higher than 94%). On the other hand, polysaccharides such as starch ethers or starches generally induce a low increase in water retention (*i.e.* up to 85%).

Mortar consistency may contribute to its water retention capacity but this hypothesis should be checked by further investigations. Indeed, cellulose ethers could induce excellent water retention thanks to the possible superposition of two phenomena [2]:

- a rheological effect similar to the one caused by other polysaccharides;
- an effect that could be specific to cellulose ethers, which remains to be defined. It could be caused by a modification of the porous network in the fresh state, osmotic pressure or the presence of a cellulose ether film playing the role of diffusion barrier.

The molar mass distribution of polysaccharides has been determined by size exclusion chromatography. Using such well-known molecules, it should be possible to understand the functioning of three types of cellulose ethers (HEC, HPMC and HEMC) on mortar water retention. Then, the influence of mortar consistency on water retention has been studied in order to verify if there is a relationship between these two parameters. To complete the panel, the impact of starch ethers on water retention and mortar consistency has also been investigated.

2. Materials and experimental methods

2.1. Mineral products

Mortars were prepared with the CEReM formulation which is given in Table 1. Mixing procedure was in accordance with EN 196-1 [3].

Table 1: Formulation of CEReM mortar.

<i>Component</i>	<i>Cement</i>	<i>Sand</i>	<i>CaCO₃</i>	<i>Admixture</i>	<i>Water mQ (Millipore®)</i>
<i>% wt of dry mixture</i>	30 %	65 %	5 %	0.27 %	30 %
<i>Amounts for 2 kg of mortar</i>	460.6 g	997.9 g	76.8 g	4.15 g	460.6 g

Sand was supplied by SIFRACO[®], its reference is DU 0.1 / 0.35. The filler reference (CaCO₃) is BL200 from OMYA[®].

Grey Portland cement was CEM I 52.5 N according to EN 197-1 [4], supplied by LAFARGE. Chemical analysis was carried out by X-ray fluorescence (XRF) spectroscopy. This technique enables to determine oxides cement-composition. Then, phase compositions were calculated using Bogue formula [5]. The analysis was performed both by X-Ray Diffraction (XRD) analysis, and using Rietveld quantification (Siroquant V2.5 software). The composition of the cement is given in Table 2.

Table 2: Chemical and phase composition of the investigated cement.

Chemical composition (% wt)		Phase composition (% wt)		
<i>Oxides</i>	<i>XRF</i>	<i>Phases</i>	<i>X-ray fluorescence (Bogue)</i>	<i>X-ray diffraction (Rietveld)</i>
CaO	66.3 ± 0.2	C₃S	64.3 ± 0.8	67.9 ± 1.2
SiO ₂	22.3 ± 0.1	C₂S	15.5 ± 0.3	15.8 ± 0.8
Al ₂ O ₃	3.40 ± 0.01	C₃A	4.2 ± 0.1	3.8 ± 0.4
SO ₃	3.04 ± 0.03	C₄AF	8.7 ± 0.1	9.4 ± 0.3
Fe ₂ O ₃	2.87 ± 0.03	Sulfates	3.04 ± 0.03	3.1 ± 0.3
MgO	0.99 ± 0.01			
P ₂ O ₅	0.24 ± 0.01			
TiO ₂	0.18 ± 0.18			
K ₂ O	0.04 ± 0.04			
MnO	0.016 ± 0.001			

Every experiment was carried out three times to make smaller uncertainties in measurement. For both methods (XRF and XRD), cement phase composition results are similar.

2.2. Organic admixtures

Admixtures are especially formulated products which are added in small amounts to mortar during the mixing process in order to modify its properties.

2.2.1. Cellulose ethers

Cellulose, the most abundant polymer in nature, forms a part of polysaccharides family derived from β-D-glucopyranose. Cellulose ethers are obtained by alkalization or alkylation of cellulose. In keeping with the substitution, different cellulose ethers can be obtained. In this

study, three kinds of cellulose ethers will be studied (Figure 1): hydroxypropylmethyl cellulose (HPMC) (a), hydroxyethylmethyl cellulose (HEMC) (b) and hydroxyethyl cellulose (HEC) (c).

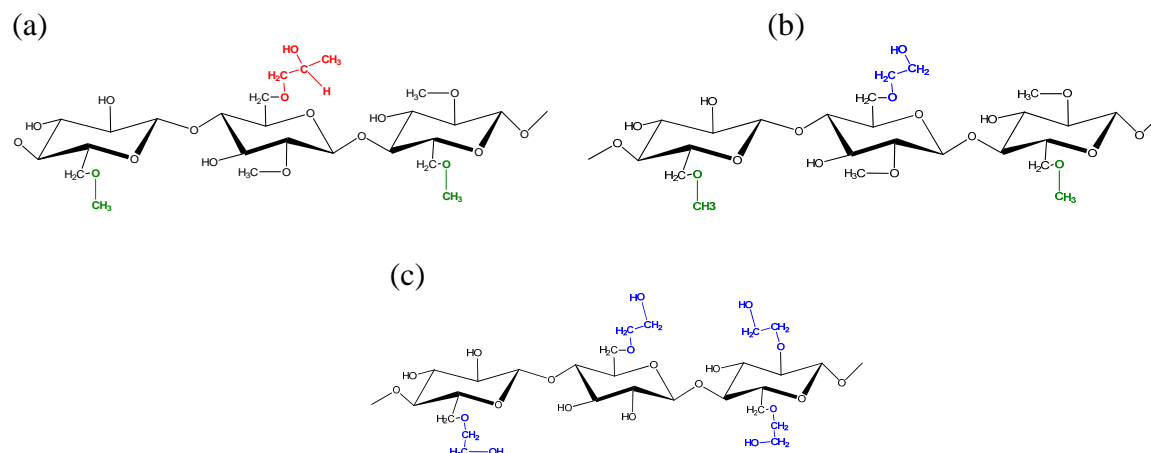


Figure 1: Structure of cellulose ethers (a: HPMC, b: HEMC, c: HEC).

On Figure 1, substituent positions are arbitrary; they differ from a molecule to another. The panel is composed of 21 molecules (Table 3):

- 3 HPMC (named A);
- 4 HEMC (named C);
- 14 HEC (named H, and N);

These products play an important role in foods, cosmetics, pharmaceuticals, latex paints, construction products, ceramics, and a host of other applications. In building materials, cellulose products are used as thickeners, binders, film formers, and water-retention agents.

HEMC	<i>Mp</i> (x1000 Daltons)	<i>Méthoxyl group</i> (% OCH₃)	<i>DS</i>	<i>Hydroxyethyl group</i> (% OC₂H₄OH)	<i>MS</i>
C1	90	28.4	1.8	4.7	0.15
C2	180	27.4	1.7	4.8	0.15
C3	310	27.4	1.7	4.8	0.15
C4	380	27.4	1.7	4.8	0.15

HPMC	<i>Mp</i> (x1000 Daltons)	<i>Méthoxyl group</i> (% OCH₃)	<i>DS</i>	<i>Hydroxypropyl group</i> (% OC₃H₆OH)	<i>MS</i>
A1	50	28.0	1.9	9.5	0.35
A2	80	21.5	1.4	9.5	0.33
A3	220	28.0	1.9	9.5	0.35

<i>HEC</i>	<i>Mp</i> (<i>x1000 Daltons</i>)	<i>Hydroxyethyl group</i> (% <i>OC₂H₄OH</i>)	<i>MS</i>
H1	45	45.3	1.9
H2	120	46.9	2.0
H3	275	49.8	2.2
H4	430	51.2	2.3
H5	720	52.5	2.4
H6	770	52.5	2.4
H7	790	52.5	2.4
N1	40	56.0	2.5
N2	630	56.0	2.5
N4	1 100	56.0	2.5
N7	1 500	56.0	2.5
N3	2 200	56.0	2.5
N5	2 300	56.0	2.5
N6	2 900	56.0	2.5

Table 3: Cellulose ethers used in this study.

2.2.2. Starch ethers

Starch is a naturally occurring high-polymeric carbohydrate composed of glucopyranose units joined together by alpha-glucosidic linkages. Starch occurs in a white granules form, usually made up of a linear polymer (amylose, Figure 2.a) and a branched polymer (amylopectin, Figure 2.b).

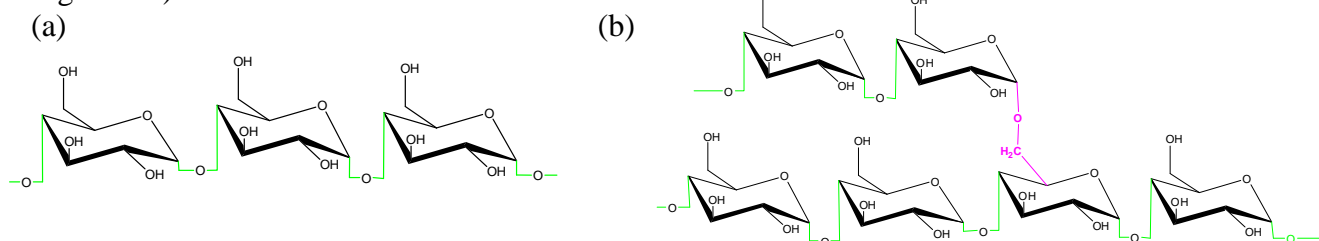


Figure 2: Structure of starch (a: amylose, b: amylopectin).

Starch ethers are obtained by the reaction of alkyl groups with etherifying agents.

In this study, seven starch ethers were used. M1 and M4 are two carboxymethyl-hydroxypropyl starches, and M2, M3 and L2 are three hydroxypropyl starches. These five compounds have an amylopectin / amylose ratio equal to 80 / 20, and their polymerization degree are respectively 2 000 000 and 4 000. L1 and L3 are two modified starches.

Starch ethers lead to better workability and improve application properties of the fresh mortar.

2.2.3. Characterization of admixtures

All polysaccharides were analysed by Size Exclusion Chromatography (SEC) in order to obtain their average molecular weights. The benefits of SEC are the low sample quantities, a wide separating range ($10^3 - 10^7$ g/mol) and short analysis time (1h) [6]. With this chromatographic method, the particles are separated based on their size. The SEC principle is that the particles of different sizes will elute through a stationary phase at different rates. The larger the particles, the faster the elution. SEC analysis was performed on a Waters apparatus

equipped with a pump (Waters 916) and a refractometer-type detector (Waters 410). The specific column used for SEC of polysaccharides is a TOSHAAS TSK Gel GMPWXL column. In our situation, the eluent was a 0.5 mol/L sodium chloride solution in order to avoid agglomeration. The eluent was filtered and in line-degassed; the flow was set to 0.5 mL/min. The column was kept at 35 °C in a furnace.

A calibration was performed using standards with known molecular weight and a theoretical polydispersity index close to 1. Height polymaltotrioses SHODEX standard P-82 were used, which are linear polysaccharides with a chemical structure close to the cellulose ethers one. Their molecular weight ranging from 5 800 Daltons (P5) to 788 000 Daltons (P800) and their retention time are, respectively 16.10 minutes and 11.98 minutes. The obtained peaks allow to establish the calibration curve by drawing $\ln(M_w)$ *versus* the retention time. Subsequently, every chromatogram was divided into many slices of 0.15 secondes in width and h_i in height. Thanks to the calibration curve, every retention time corresponds to a mass M_i of N_i molecules eluted of the studied polymer. The height of every slice is directly proportional to the concentration c_i of the eluted polymer. Therefore, the weight-average molecular mass (noted \overline{M}_w) and the number-average molecular mass (noted \overline{M}_n) were calculated thanks to the following equations 1a and 1b.

Equation 1 : Formula to calculate the weight-average (a) and the number-average (b) molecular mass

$$(a) \quad \overline{M}_w = \frac{\sum c_i \cdot M_i}{\sum c_i} \qquad (b) \quad \overline{M}_n = \frac{\sum c_i}{\sum \frac{c_i}{M_i}}$$

2.3. Water retention measurements

Water retention is a mortar property that prevents the rapid loss of water to substrate by suction. This property prevents bleeding or “water gain” when the mortar is in contact with relatively impermeable units. Water retention is a fundamental property and affects workability and bond between mortar and masonry units. Water retention depends on mortar formulation.

According to the DTU 26.1 [7], mortars are divided into three classes:

- the first one is for mortars which have a water retention lower than 86 %. They belong to the *low* water retention category;
- the second class (*intermediate* water retention): when the value is between 86 % and 94 %;
- and when water retention is higher than 94 %, it is a *strong* water retention mortar

These limits have to be used with care because they refer only to the ASTM C91 measurements.

In this study, water retention measurements were performed using two different standards: the DIN 18555 and the ASTM C91 methods. Experiments were carried out three times for each material.

2.3.1. Water retention using DIN 18555

The standard DIN [8] was carried out **5 minutes** after mixing. It consists in measuring the lost water of a mortar in contact with a filter plate within a period of **5 minutes**. The mortar was poured into a conical ring previously placed on a filter paper, as shown in Figure 3. The assembly was weighed, covered with a plastic plate and left to stand for 5 minutes. By weight differences, the water retention WR (in %) was thus deduced using equation 2.

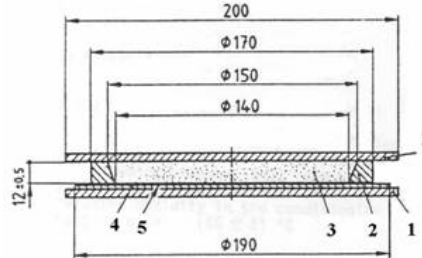


Figure 3: Test device for standard DIN 18555. 1: plastic plate; 2: plastic ring; 3: fresh mortar; 4: filter paper; 5: non woven tissue. Dimensions are in mm.

Equation 2 : Formula to calculate the water retention capacity using DIN 18555 standard.

$$WR \text{ (\%)} = 100 - \frac{m_7 - m_6}{(m_5 - m_4) \times W_1} \times 100$$

Where :

- m_4 : (plastic plate + dry filter plate) + nonwoven tissue + plastic ring
- m_5 : (plastic plate + dry filter plate + nonwoven tissue + plastic ring) + mortar
- m_6 : plastic plate + dry filter plate
- m_7 : plastic plate + soaked filter plate
- W_1 : percentage of water in the mortar (23% for CEREM formulation)

2.3.2. Water retention using ASTM C91

Concerning the standard ASTM C 91 [9], measurements have to be performed **15 minutes** after mixing. The aim of the test is to measure the water loss of a mortar under a depression of 50 mmHg for **15 minutes** (Figure 4).

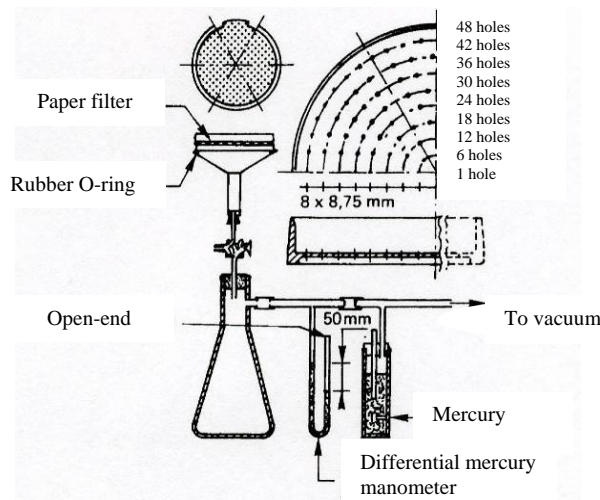


Figure 4: Experimental device to measure water retention with standard ASTM C91. Then, the water retention capacity is calculated using equation 3.

Equation 3: Formula to calculate the water retention capacity using ASTM C91 standard.

$$WR \ (%) = \frac{W_0 - W_1}{W_0} \times 100$$

Where:

- W_0 represents the initial mass of mixing water
- W_1 is the loss of mass of mixing water after 15 minutes under depression.

2.4. Consistency

Workability is an essential property of any mortar used in construction. This property improves the contact between mortar and substrates.

This parameter could be measured using CSTB method (Consistor Baronnie) [10]. This method was chosen because it is very easy and quick to set up; however, results need to be confirmed with a more rigorous method (rheological measurements).

The material consists of ten stainless steel tubes with diameter ranging from 10 to 55 mm; they are marked from 1 to 10 (Figure 5a).

(a)



(b)



Figure 5: Test devices to measure mortar consistency.

The tube is tight inside the dish (Figure 5b), filled with mortar and levelled off. The shutter of the dish is quickly opened and the mortar falling time is measured. The aim of the experiment is to find the largest tube for which the paste does not slide before 5 seconds. The consistency of the paste is given by the number which corresponds to the largest tube. If the consistency is not measurable, the result will be noted f (for “fluid”). Measurements were performed with the same batch as water retention one.

3. Results and discussion

3.1. Correlation between ASTM C91 and DIN 18 555 test methods

For the whole study, water retention measurements are performed with both methods. The comparison between both methods of measurement is shown on figure 6.

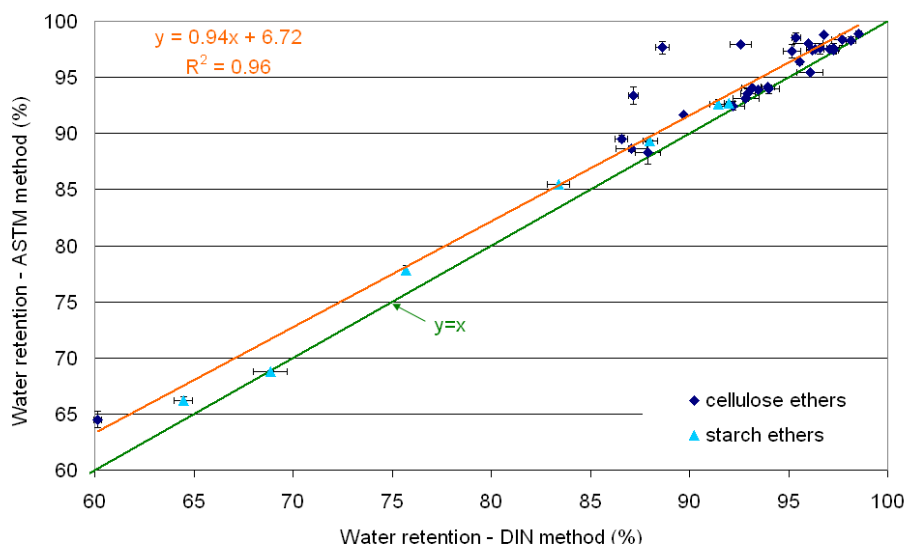


Figure 6 : Comparison between the DIN and the ASTM method.

Usually, both methods give rather close results, except for a few molecules. Among these are three HEC (N1, N2 and N3). These additives conferred to the mortar a more liquid aspect. The water retention value obtained with the DIN method is close to 90 % while the result of ASTM is considerably higher (roughly 97 %). This could be explained by a bleeding phenomenon observed for these admixtures during the experiment. Indeed, water seems to stay above whereas sand is at the bottom.

Furthermore, the values obtained with the ASTM method are usually higher than those obtained with the DIN method (the linear regression is over the first bisecting line). This phenomenon could be explained by a depression effect. Indeed, the ASTM measurement is carried out with a depression of 50 mmHg while the DIN method is a measurement of absorption of water in contact with a paper filter, based on gravitation and performed at atmospheric pressure.

All things considered, the DIN 18555 and the ASTM C91 methods are two ways of determining water retention which are roughly comparable.

3.2. Influence of molecular mass on the water retention capacity

3.2.1. Results for HEMC

To study the weight-average molecular mass influence, the sample group C was chosen. Indeed, these compounds have a constant chemical structure except for C1 which has a slightly different methoxyl percentage (Table 4).

Table 4: Range of HEMC to investigate the impact of \overline{M}_w .

<i>Admixtures HEMC</i>	<i>Méthoxyl (% OCH₃)</i>	<i>Hydroxyethyl group (% OC₂H₄OH)</i>	\overline{M}_w (x1000 Daltons)
<i>C1</i>	28.8	4.7	90
<i>C2</i>	27.4	4.8	180
<i>C3</i>	27.4	4.8	310
<i>C4</i>	27.4	4.8	380

For a constant chemical structure, experimental results of water retention highlight the impact of admixture molecular weight (Figure 7).

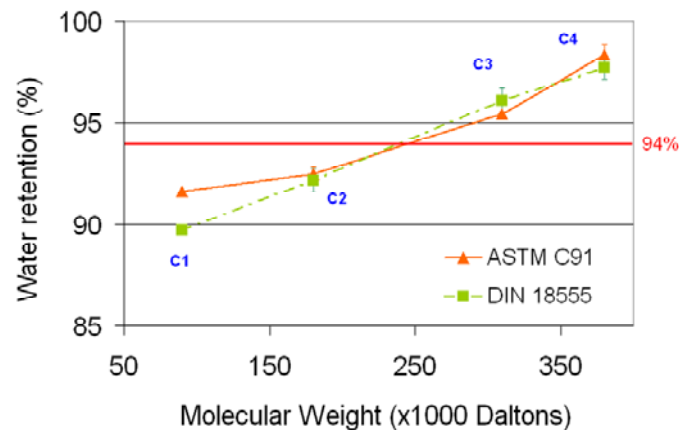


Figure 7: Influence of molecular mass of HEMC on water retention.

In spite of C1 having a slightly different methoxyl percentage, this result is in line with the three other HEMCs. Hence, for HEMC having a molecular mass lower than 400 000 Daltons, the higher the molecular mass, the better the mortar water retention capacity. Moreover, two of these four admixtures do not procure strong water retention (C1 and C2). For low HEMC molecular masses, the mortar water retention belongs to the intermediate class defined by the DTU. On the contrary, C3 and C4 give a strong water retention capacity to the mortar.

Consistency measurements for HEMC C group can also be related to the cellulose ether molecular mass (Figure 8).

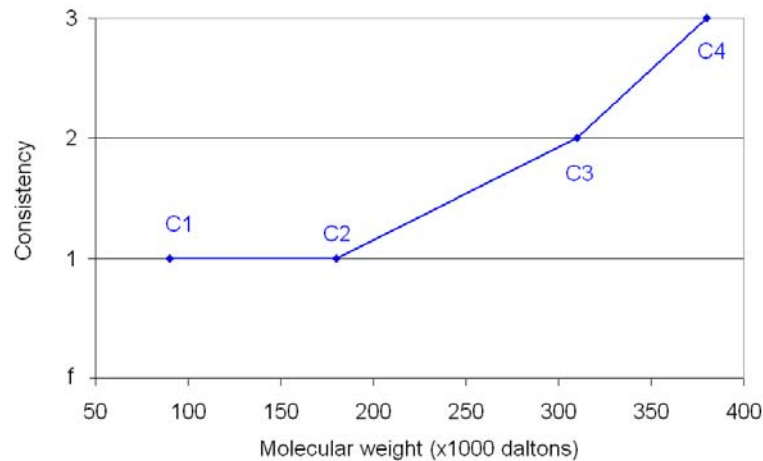


Figure 8: Influence of molecular mass of HEMC on mortar consistency.

For this HEMC sample group, both the mortar consistency and water retention increase with admixture molecular mass. A hypothesis can be emitted: when the HEMC molecular mass increases, the mortar consistency increases too, which has itself an impact on the water retention. This can be explained by the HEMC capacity to form, with mixing water, a more or less viscous gel. As a consequence, a high molecular mass admixture would decrease the water mobility (due to this gel) with the result that the water retention would be improved.

For the HEMC C, the higher the molecular mass, the better the consistency and the water retention capacity of the admixed mortar.

3.2.2. Results for HPMC

Among HPMC, only two cellulose ethers (HPMC A) have a constant chemical structure (Table 3). The HPMC molecular mass influence can be studied using these two molecules. The experimental results of water retention and consistency are given in the Table 5.

Table 5: Range of HPMC to investigate the impact of Mw.

<i>Admixtures HPMC</i>	<i>Mw (x1000 Daltons)</i>	Water retention (%)		<i>Consistency</i>
		DIN	ASTM	
<i>A1</i>	50	(86.6 ± 0.3)	(89.5 ± 0.3)	1
<i>A3</i>	220	(92.9 ± 0.4)	(93.6 ± 0.4)	2

The observed trend is the same as for HEMC. The higher the HPMC molecular mass, the better the water retention capacity and the mortar consistency. However, it is difficult to conclude with only two molecules. This study needs to be widened using compounds with intermediate and higher molecular masses.

3.2.3. Results for HEC

Admixtures from group N present a wide range of molecular mass ranging from 40 000 to 2 900 000 Daltons. According to the manufacturers' data, the hydroxyethyl percentage is equivalent for all these cellulose ethers (56 %). In this sample group, only one parameter varies: the HEC molecular mass. Even though absolute water retention results differ between DIN and ASTM methods, the trends are similar (Figure 9).

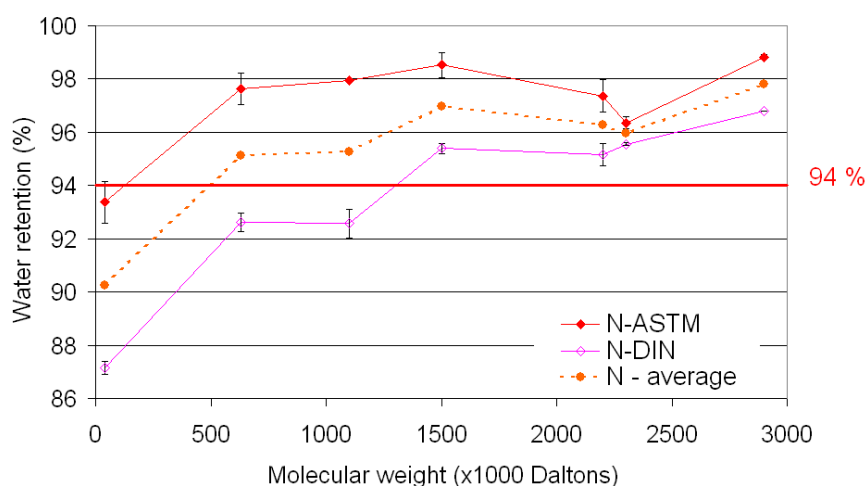


Figure 9: Influence of molecular mass of HEC N on water retention.

For HEC with molecular masses ranging from 40 000 to 630 000 Daltons, mortar water retention varies from 87 % to 92 % for standard DIN and from 93 % to 97 % for standard ASTM. For HEC with molecular masses ranging from 630 000 to 2 900 000 Daltons, water retention rise is less important (ranging from 92 % to 96 % for standard DIN and from 97 % to 98 % for standard ASTM).

The water retention increases with the HEC molecular mass up to a threshold value. This value can be estimated at roughly 600 000 Daltons. However, it is difficult to conclude because there are many intermediate molecular masses between 40 000 and 630 000 Daltons. From this value, the molecular mass seems to influence only slightly the mortar capacity to retain water. This trend is confirmed with the results obtained for another HEC family: H (Figure 10). However, among this panel, the substitution degree is also varying from 45 % to 52.5 % (respectively for H1 and H2).

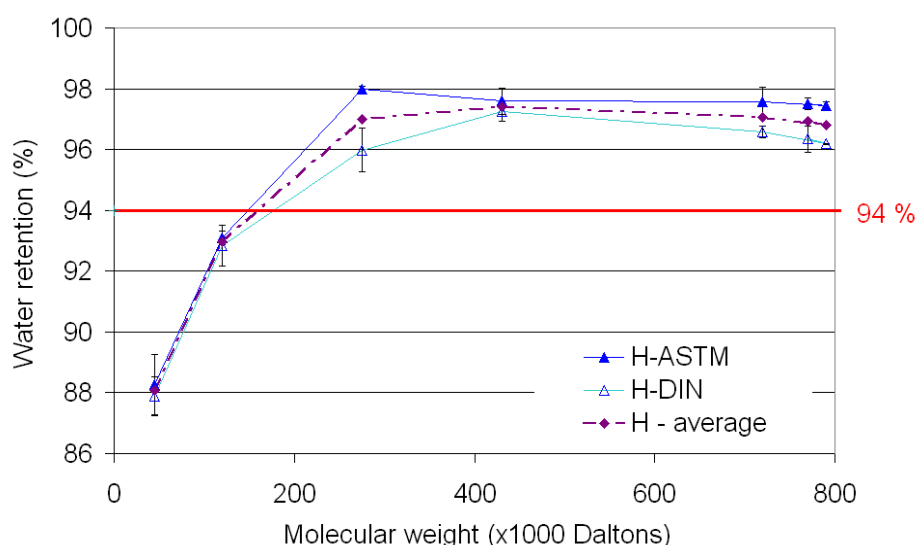


Figure 10: Influence of molecular mass of HEC H on water retention.

With this admixtures H, a plateau is observed from 275 000 (H3) to 790 000 Daltons (H7). In this range, mortar water retention is slightly constant whatever the HEC molecular mass. In consequence for HEC with a hydroxyethyl percentage ranging from 49.8 to 52.5 %, the molecular mass does not have any influence on water retention from 275 000 Daltons.

Regarding H1 and H2, in comparison with the complete panel, these admixtures have two parameters varying (molecular mass and substitution degree). The conclusion therefore is difficult to establish. However, the trend is in line with the HEC N. There is a threshold value from which the molecular mass does not influence the mortar water retention.

The relationship between mortar consistency and HEC molecular mass is similar to the water retention evolution (Figure 11).

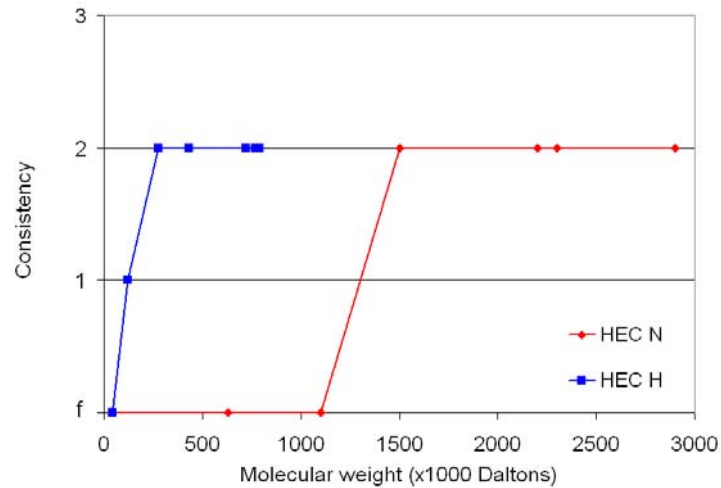


Figure 11: Influence of molecular mass of HEC N and H on mortar consistency.

For these two admixtures panels, the consistency was not measurable for all mortars because they looked like the non-admixed mortar. Nevertheless, in each sample group, mortar consistency is better for high HEC molecular weights.

3.3. Impact of mortar consistency on water retention capacity

In part 3.2.1 a hypothesis was formulated concerning the influence of mortar consistency on water retention capacity. Indeed, for HEMC C, water retention increases when mortar consistency increases. However, the correlation between water retention and consistency does not seem to be so obvious. Some starch ethers were studied and these compounds procure very poor water retention to the mortar while paste consistency is pretty good (Figure 12).

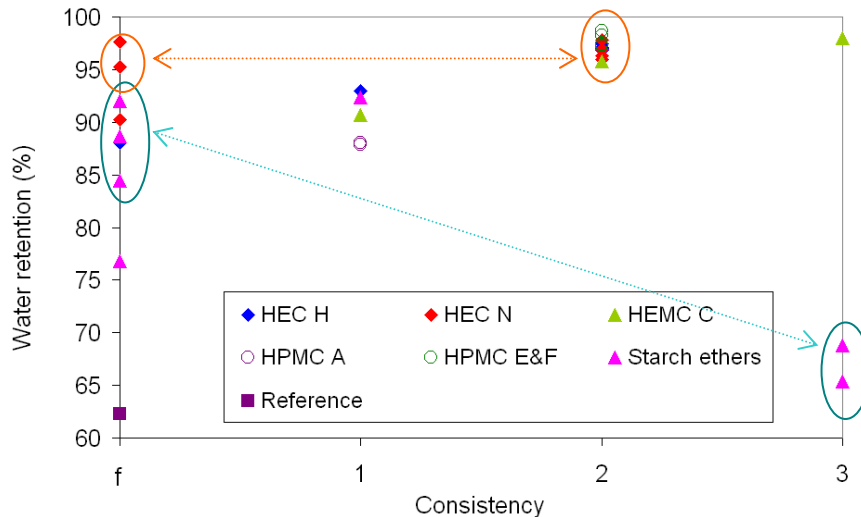


Figure 12: Evolution of water retention as a function of mortar consistency.

These results show that HPMC and HEMC usually procure strong water retention and pretty good consistency for the mortar. However, some HEC (notably two molecules from group N) bring strong water retention (*i.e.* higher than 95 %) to the fresh mortar, whereas the paste consistency is close to the non-admixed one. Furthermore, admixed mortar can have different consistencies ranging from unmeasurable to 2, whereas the same water retention can be observed. As a consequence, a major conclusion of this study is that the water retention capacity of a fresh mortar is not only due to its consistency.

4. Conclusions and new insights

Gradual effects on mortar water retention are clearly observed as a function of cellulose ethers chemistry. Therefore, one of the main conclusions of this study is that the polymer chemistry (HEMC, HPMC, or HEC) and the structural parameters (particularly molecular mass) are important factors controlling water retention and mortar consistency. For HEMC and HPMC, consistency and water retention increase with cellulose ether size. On the other hand, for HEC, a threshold value for molecular mass was observed. Different thresholds were noticed between two families with different substitution degrees. However, the studied panel does not allow us to conclude about the impact of substitution degree on water retention.

This study also demonstrates that mortar water retention is not only a function of its consistency. Nevertheless, consistency results have to be confirmed with a rigorous method. Measurements of yield stress with a controlled shear stress rheometer supplied with a helical geometry may be an interesting way to characterize mortar consistency.

For a better understanding of the functioning of cellulose ethers on water retention mechanism, two parameters will be studied. First, the polymer particle size will be explored by performing water retention measurements with different particle size grades. It will be taken to think that the thinner the admixture particle, the better the polymer rate dissolution. Secondly, mortar water retention capacity will be studied as a function of cellulose ether concentration in mortar formulation. Indeed, Afridi [11] using different kind of admixtures (latex) showed that water retention of polymer-modified mortars increases with a rise in polymer-cement ratio.

Water retention may be explained by an effect of barrier diffusion of cellulose ethers film. Polymer films in a freshly mixed mortar were observed by Jenni [12]. This behaviour was particularly studied for a HPMC; in consequence, this property should be carried out whatever the cellulose ether molecule. Moreover, cellulose ether capacity to form film could be studied using a polarized-light microscope.

5. References

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